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Dipole moments and conformational analysis of tris(2-pyridyl)phosphine and tris(2-pyridyl)phosphine chalcogenides. Experimental and theoretical study

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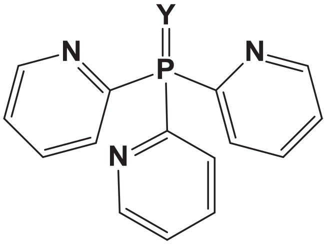
HIGHLIGHTS

- Dipole moments of tris(2-pyridyl) phosphine and its chalcogenides were determined.
- Possible conformers of phosphines were optimized using density functional theory.
- Conformational analysis of tris (2-pyridyl)phosphine and its chalcogenides was performed.
- Tris(2-pyridyl)phosphine and its chalcogenides have preferred *gauche*- and *trans*-forms.

GRAPHICAL ABSTRACT



Y = lone pair **1**, O **2**, S **3**, Se **4**

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ABSTRACT

Conformational analysis of tris(2-pyridyl)phosphine and tris(2-pyridyl)phosphine chalcogenides was carried out by the method of dipole moments and density functional theory calculations. The conformations of the examined compounds fit into the overall conformational picture for the P^{III} and P^{IV} compounds: namely, these phosphines have non-eclipsed *gauche*- and *trans*-forms with propeller arrangement of the pyridyl radicals about the P = Y bond (Y = lone pair, O, S, Se).

Introduction

Extensive utilization of organophosphorus compounds for practical purposes prompted the progress in the field of their conformational analysis and necessitated synthesis of compounds with predefined useful properties, and identification of their structure and fine conformational features. Structural and polarity data are necessary for studying of reaction mechanisms and reactivity of organophosphorus compounds. For complex polyfunctional organoelement compounds in general and for organophosphorus compounds with several axes of internal rotation in particular, the majority of articles usually describes the mutual arrangement of separate fragments of molecules established without conformational analysis of an entire molecule as a whole. In this study, we used a comprehensive approach to investigate the structure of molecules in solution using various complementary physical methods taking into account all their advantages and/or

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disadvantages. This methodology is exceptionally effective for the investigation of diverse classes of compounds in solution [1]. The importance of this work can hardly be exaggerated since the majority of chemical reaction occurs in solution. To our knowledge, this is one of very few such studies of organophosphorus compounds.

Tris(2-pyridyl)phosphine and its chalcogenides have attracted considerable attention due to their applications as ligands for the design of metal-complex catalysts of such important industrial reactions as methoxycarbonylation of alkynes, hydroformylation of alkenes, ethylene polymerization, and Diels–Alder synthesis [2–5]. The recently developed novel one-pot synthesis of tris(2-pyridyl)phosphine from 2-bromopyridine and elemental phosphorus [6] creates broad opportunities for further development of chemistry of this promising functional phosphine and its derivatives. In the present research, tris(2-pyridyl)phosphine and tris(2-pyridyl)phosphine chalcogenides were used as appropriate models to obtain novel fundamental data on conformational structure of the organic compounds of tri- and tetra-coordinated phosphorus atom.

Experimental

Materials and physical measurements

Tris(2-pyridyl)phosphine **1** and oxide **2** were synthesized according to procedures [6,7], sulfide **3** and selenide **4** were prepared from phosphine **1** and elemental sulfur or selene in toluene according to procedure [8]. The solvents were purified using standard procedure.

The NMR ^{31}P spectrum was registered on a Bruker Advance III spectrometer (400 MHz). The IR spectra were registered on a Bruker Vector 22 spectrometer.

Dipole moments

The experimental values of the dipole moments were determined according to the second Debye method. Physical parameters of **1–4** were measured from series consisting of 4–6 solutions in dioxane at 25 °C. The dielectric permittivity of solutions of **1–4** was determined on a BI-870 instrument (Brookhaven Instruments Corporation), the accuracy is ±0.01. The refractive indices of solutions were determined on a RA-500 refractometer (Kyoto Electronics), the accuracy is ±0.0001.

The experimental dipole moments were calculated by the formula [9]:

$$\mu = 0.01283\sqrt{P_{\text{or}}T}.$$

The orientation polarizabilities P_{or} were calculated by the Guggenheim–Smith formula [10,11]:

$$P_{\text{or}} = \frac{M}{d}\left[\frac{3\alpha}{(\varepsilon_0+2)^2} - \frac{3\gamma}{(n_0^2+2)^2}\right],$$

where M is the molecular weight of a substance, d is the density of the solvent, α and γ are the slope tangents of the straight lines in the coordinates ε_i–w_i and $n_i^2 - w_i$; and ε_i, n_i, and w_i are, respectively, the dielectric permittivity, refractive index, and weight fraction of the solute in ith solution. The coefficients α and γ were calculated by the formulas: $\alpha = (\varepsilon_i - \varepsilon_0)/\omega_i$ and $\gamma = (n_i^2 - n_0^2)/\omega_i$, where ε_0 and n_0 are, respectively, the dielectric permittivity and refractive index of the solvent.

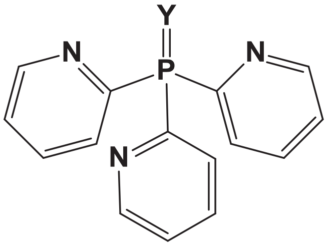
In the calculations of dipole moments according to the vector-additive scheme we used the following bonds moments: $m(\text{P=O}) = 2.94$ D calculated from μ_{exp} $\text{Ph}_3\text{P=O}$ [12], $m(\text{P=S}) = 3.29$ D calculated from μ_{exp} $\text{Ph}_3\text{P=S}$ [12], $m(\text{P=Se}) = 3.34$ D calculated from μ_{exp} $\text{Ph}_3\text{P=Se}$ [13], $m(\text{C}_{\text{sp2}} \to \text{P}) = 0.39$ D [12].

Quantum chemical calculations

Quantum chemical calculations were carried out using density functional theory (DFT) method B3LYP with wB97XD/6-311++G(df,p) basis set by the GAUSSIAN 09 program [14] with the total optimization of geometry. Correspondence of the obtained stationary points to the energy minima was proved by calculation of the second derivatives in all cases. All calculations were performed in Kazan Branch of Joint Supercomputer Center of the Russian Academy of Sciences (http://wt.knc.ru).

Results and discussions

In this study, we have determined previously unknown polarities of tris(2-pyridyl)phosphine **1**, its oxide **2**, sulfide **3**, and selenide **4**, carried out quantum chemical calculations of the dipole moments of their possible conformers, and calculated their polarities according to the vector-additive scheme.



Y = lone pair **1**, O **2**, S **3**, Se **4**

It should be mentioned, that the method of dipole moments is a sensitive instrument for the determination of structure and the study of fine features of spatial and electronic structure of polar organic and organoelement compounds in solution. For determining the experimental values of the dipole moments we used the second Debye method based on the measurement of the dielectric constant of the dilute solutions of the polar substance in a nonpolar solvent [9]. The experimental dipole moments of **1–4** are listed in Table 1, their values are sufficiently high.

We noticed the unexpectedly great value of experimental dipole moment of phosphine **1** (4.16 D). Usually, the experimental dipole moments of the majority of three-valence phosphorus compounds do not exceed 3 D [12]. We registered the NMR ^{31}P spectrum of phosphine **1** under conditions of the experiment for dipole moments, and the spectral result (singlet $\delta = -1.44$ ppm, dioxane) was practically identical to the literature data ($\delta = -1.98$ ppm) [7], indicating that **1** does not undergo any changes under these conditions. The follow-up study supported the finding that the polarity of tris(2-pyridyl)phosphine is relatively high.

Table 1
Coefficients in the calculation equations, orientation polarizabilities, and experimental dipole moments of **1–4**.

Compound	α	γ	P_{or} (cm^3)	μ (D)
1	8.751	0.547	358.154	4.16
2	8.973	0.449	395.090	4.37
3	6.971	0.593	309.694	3.87
4	7.003	0.543	363.479	4.19

First, the DFT B3PW91/6-31G(d) method was used to identify possible conformations of the studied compounds. We examined all possible conformations, which appear as a result of rotation about the single bonds P–C, and selected those forms, which had minimum relative energy. Then, the precise calculations using the broadened basis set 6-311++G(df,p) were performed. However, the calculation by the B3PW91/6-311++G(df,p) method gave slightly different results, specifically, the conformations having the energy minimum differed from those with energy minimum according to the B3PW91/6-31G(d) method. Accordingly, we decided to use the more modern DFT wB97XD method which includes the improved exchange functional and takes into account the dispersion interactions within a molecule [15].

Previously, we have already tested this method in combination with the broadened basis set 6-311++G(df,p) [16]: it gives slightly better agreement between the computed and experimental values of dipole moments, although it demands appreciably greater calculation time. In the present case, the computed value of dipole moment of the model compound triphenylphosphine (1.55 D) practically coincides with the reference value – 1.50 D [17]. In general, these results are consistent with the data obtained by the B3PW91/6-311++G(df,p) method, so in this paper we only provide the wB97XD/6-311++G(df,p) results considering them more reliable.

We identified the possible conformations of isolated molecules of **1**–**4** using the wB97XD/6-311++G(df,p) method, calculated their theoretical polarities and relative energies, and compared theoretical results with experimental data.

The possible conformers of **1**–**4** are shown in Figs. 1, 2, 4 and 5, while their relative energy values and theoretical dipole moments are listed in Table 2.

According to the quantum chemical calculations tris(2-pyridyl)phosphine, its oxide, sulfide, and selenide have the structure of a pyramid in which the pyridyl cycles arrange like a propeller about the P = Y bond (Y = lone pair, O, S, Se). We did not take into account the computed mirror isomers with the same values of relative energy and dipole moments.

Two conformations of unsubstituted tris(2-pyridyl)phosphine **1** corresponding to the energy minimum are possible (Table 2, Fig. 1): **1a** – the nitrogen atoms of all pyridyl cycles are directed towards the lone pair of electrons at the P atom, and **1b** – two pyridyl cycles are directed towards the lone pair of electrons at the P atom by the nitrogen atoms. The dihedral angles *cis*-lone pair-P-C_{sp2}-N is equal to 50–60° which corresponds to *gauche*, *gauche*,*gauche*-orientation of the pyridyl cycles under rotation about the P-C_{sp2}(cycle) bond. Judging from the correlation of the theoretical and experimental dipole moments, there is a conformational equilibrium of **1a** and **1b** in solution.

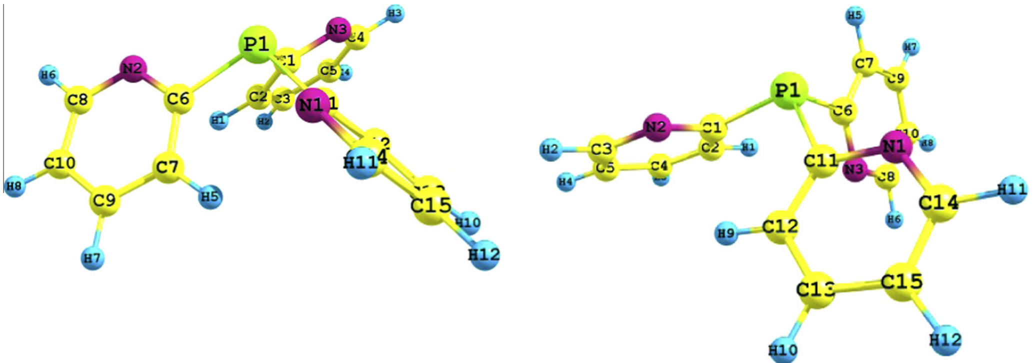
According to the data of X-ray diffraction [18], tris(2-pyridyl)phosphine **1** is pyramidal in the crystal state as well, but the conformation of the molecules in the crystal **1** is different from the calculation results: one of the nitrogen atoms is directed to the same side of the molecule as the P^{III} lone pair of electrons, while the remaining two N atoms point in the opposite direction. The phosphorus atom is almost coplanar with each of the rings.

For tris(2-pyridyl)phosphine oxide (Fig. 2, Table 2), there are three possible conformations: **2a** – all three nitrogen atoms of pyridyl cycles are directed away from the P = O bond (dihedral angles O = P-C_{sp2}-N are about 144° which correspond to *trans*,*trans*,*trans*-orientation); **2b** – only one pyridyl cycle is directed by the nitrogen atom towards the P = O bond (dihedral angles O = P-C_{sp2}-N are 86°, 176°, 164° which correspond to *gauche*,*trans*,*trans*-orientation); and **2c** – all three nitrogen atoms of pyridyl cycles are directed towards the P = O bond (dihedral angles O = P-C_{sp2}-N are about 56° which correspond to *gauche*,*gauche*,*gauche*-orientation). In contrast to tris(2-pyridyl)phosphine, conformation **2c** becomes the most energetically disadvantageous.

Because of insufficient solubility of **2** in nonpolar solvents, it is impossible to judge about its conformation by the value of its experimental dipole moment even using the minimum concentration of solutions (0.0035 mol L^{-1}). We have registered the IR spectra of phosphine oxide **2** in melt and solid state of the sample (Fig. 3). There is no change (freezing) of the band number in the IR spectra of **2** (ν P = O 1242 cm^{-1}) under the transition from the melt to solid state, indicating the absence of conformational heterogeneity of **2** in the crystal and melt states.

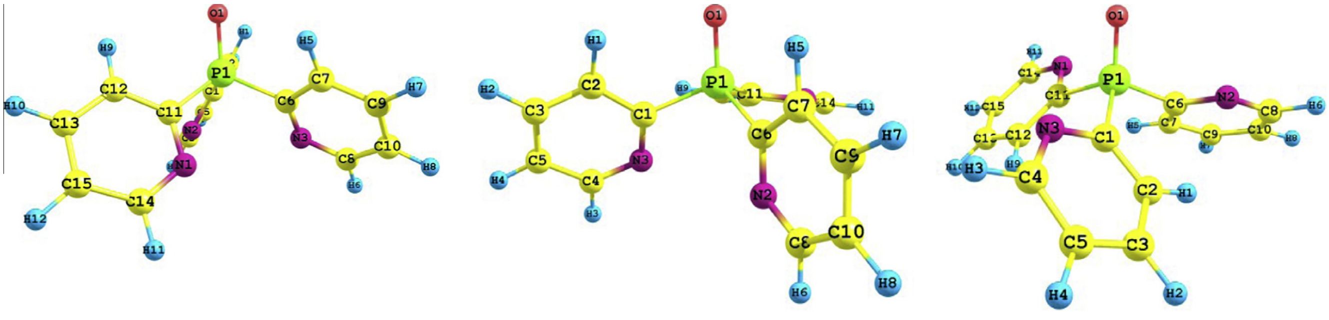
According to the data of wB97XD/6-311++G(df,p) method for the **2a** and **2b** conformers, the theoretical frequencies of the stretching vibrations of the P = O bond are identical and equal to 1258 cm^{-1}, which makes these conformers indistinguishable in IR spectra computationally simulated. Probably, for this reason the signs of conformational equilibrium are not observed in experimental IR spectra of phosphine oxide **2**.

The obtained results are in a good agreement with the data of [19] where the structure of tris(2-pyridyl)phosphine oxide in crystal was studied by the X-ray diffraction. It was established that phosphine oxide **2** has pyramid configuration with propeller arrangement of three pyridyl rings, one of the nitrogen atoms is directed to the oxygen atom of the P = O group, and another two nitrogen atoms are directed to the opposite side that is the conformation of the molecules in the crystal **2** is practically identical to the geometry of conformer **2b**. The molecules of **2** in crystal are bonded by the weak C–H···O and C–H···N intermolecular interactions, and the same C–H···N interactions exist in tris(2-pyridyl)phosphine **1** [19]. Most likely, these intermolecular interactions in **1** define the high value of its polarity in comparison



1a **1b**

Fig. 1. The possible conformers of **1** according to DFT wB97XD/6-311++G(df,p) method.



2a **2b** **2c**

Fig. 2. The possible conformers of **2** according to DFT wB97XD/6-311++G(df,p) method.

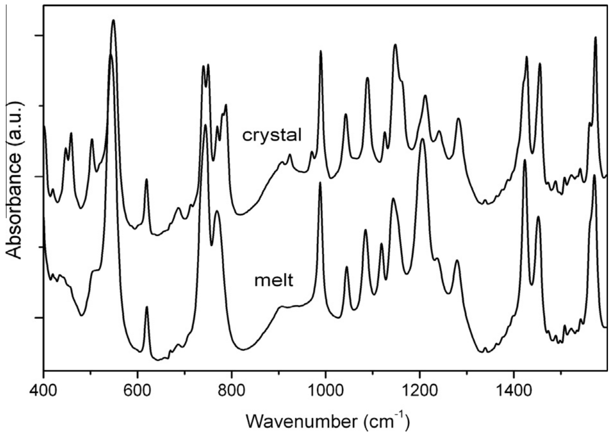


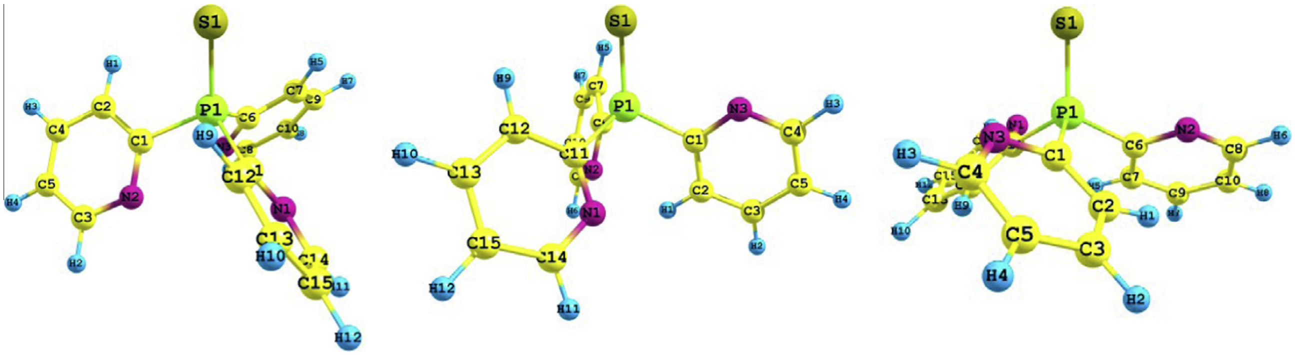
Fig. 3. FTIR spectra of tris(2-pyridyl)phosphine oxide **2** in the solid and melt states (region 400–1600 cm^{-1}).

with the majority of compounds of three-valence phosphorus [12]. It is well known that low values of dielectric permeability and, accordingly, dipole moments usually correspond to the compounds with an intramolecular H-bond, while an intermolecular hydrogen bond leads to an increase of polarity of the molecules with acyclic association [20].

Concurrence of the experimental and theoretical values of the bond distances and angles for tris(2-pyridyl)phosphine oxide **2** is very good, especially taking into account the difference between the crystal [19] and gaseous phase. The main conclusion on its conformation is also consistent between the experimental and theoretical values: namely, it is a pyramid with propeller arrangement of the pyridyl cycles, where one of the pyridyl cycles is directed by the nitrogen atom towards the oxygen atom of P = O group, and another two pyridyl cycles are directed to the opposite side.

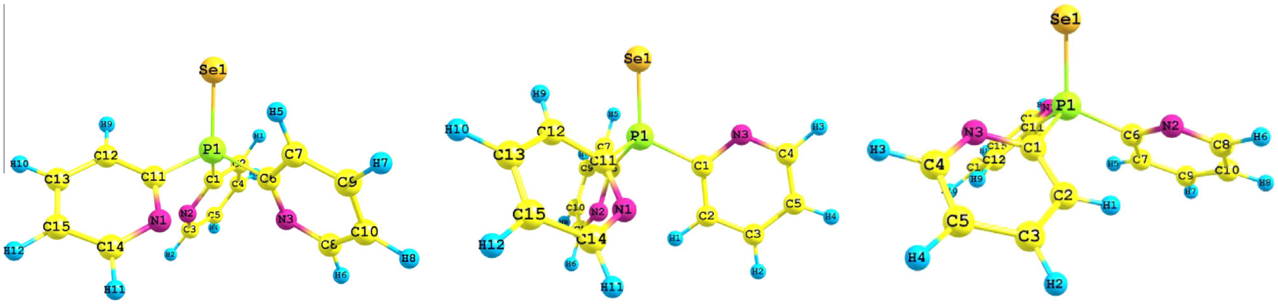
Theoretical conformations for tris(2-pyridyl)phosphine sulfide **3** and tris(2-pyridyl)phosphine selenide **4** (Figs. 4 and 5, Table 2) are analogous to the conformations of tris(2-pyridyl)phosphine oxide **2**. However, the most energetically advantageous conformations for **3** and **4** are the forms in which the nitrogen atoms are directed to the opposite side from groups P = S (**3a**) and P = Se (**4a**), that is these conformers have *trans,trans,trans*-orientation of the pyridyl cycles relative to the P = S or P = Se bond (dihedral angles S = P-C$_{sp2}$-N are about 139°; dihedral angles Se = P-C$_{sp2}$-N are about 138°). Another two possible conformers of **3** and **4** have *trans,trans,cis*-orientation of the pyridyl cycles relative to the P = S or P = Se bond (**3b** – dihedral angles S = P-C$_{sp2}$-N are 152°, 165°, 13°; **4b** – dihedral angles Se = P-C$_{sp2}$-N are 147°, 162°, 14°) or *gauche,gauche,gauche*-orientation of the pyridyl cycles relative to the P = S or P = Se bond (**3c** – dihedral angles S = P-C$_{sp2}$-N are about 56°; **4c** – dihedral angles Se = P-C$_{sp2}$-N are about 57°).

We compared the obtained results for tris(2-pyridyl)phosphine sulfide **3** and tris(2-pyridyl)phosphine selenide **4** with experimental data of single crystal X-ray analysis of them [21]. According to [21], the structures **3** and **4** are analogous to the tris(2-pyridyl)phosphine oxide [19], they have distorted tetrahedral configuration around the phosphorus atom. In crystal of phosphine sulfide **3** one nitrogen atom is directed towards the sulfur atom, and another two nitrogen atoms are directed to the opposite side. This structure is in an agreement with the structure of theoretically possible conformer **3b** (Table 2, Fig. 4). On the contrary, all nitrogen atoms in the crystal of **4** are directed towards the selenium atom, and this structure meets the energetically disadvantageous conformer **4c** (Table 2, Fig. 5) with too great a dipole moment. The



3a **3b** **3c**

Fig. 4. The possible conformers of **3** according to DFT wB97XD/6-311++G(df,p) method.



4a **4b** **4c**

Fig. 5. The possible conformers of **4** according to DFT wB97XD/6-311++G(df,p) method.

Table 2
Relative energies and theoretical dipole moments of conformers of **1**–**4**.

Conformer	ΔE (kJ mol^{-1})	μ_{theor} (D)
1a	0.0	5.61
1b	1.8	3.82
2a	3.6	2.27
2b	0.0	3.22
2c	31.1	8.41
3a	0.0	3.45
3b	8.2	5.62
3c	27.9	8.58
4a	0.0	3.64
4b	9.1	5.85
4c	26.3	8.60

molecules of **3** and **4** in the crystal cells [21] are bonded by the weak hydrogen intermolecular interactions as well as the molecules of **1** and **2** in the crystal state [19]. Structures of **1**–**4** as generally crystal structures, first of all, are determined by the requirements of crystalline packing. Probably, the weak intermolecular interactions C–H···O, C–H···S, C–H···Se or C–H···P and C–H···N in **1**–**4** promote more efficient packing. In solution, as a rule, conformational picture becomes more diverse.

Based on a comparison of the theoretical results and experimental dipole moments of these compounds, we believe that mainly a single conformer is present in solution: **2b** for tris(2-pyridyl)phosphine oxide, **3a** for tris(2-pyridyl)phosphine sulfide, and **4a** for tris(2-pyridyl)phosphine selenide with a small admixture of other conformer (minor amount of **2a** as well as **3b** and **4b**, respectively).

Using phosphine **1** as a model compound, we have calculated the group moment m(Heterocycle → P) which is equal to 3.41 D. If the models are primary pyridyl phosphine ($\mu_{\text{theor}} = 2.83$ D) or secondary bispyridyl phosphine ($\mu_{\text{theor}} = 3.26$ D), the group moments m(Heterocycle → P) are equal to 5.31 and 3.54 D, respectively. The first and third values of the group moments are close. However, the use of them in calculations of the dipole moments of phosphine oxide **2**, phosphine sulfide **3**, and phosphine selenide **4** according to the vector-additive scheme does not lead to an agreement between the experimental and calculated data. All calculated moments turned out considerably above the experimental values (by 2 D or more). It should be noted that the values of the dipole moments calculated according to the vector-additive scheme do not depend on a position of the nitrogen atom of heterocycle relative to the P = Y bond (Y = lone pair, O, S, Se).

Conclusion

We have carried out experimental and theoretical conformational analysis of tris(2-pyridyl)phosphine, tris(2-pyridyl)phosphine oxide, tris(2-pyridyl)phosphine sulfide, and tris(2-pyridyl)phosphine selenide by the methods of dipole moments and quantum chemistry (DFT calculations). For all examined compounds, the theoretical results are in a good agreement with the experimental data obtained.

In general, we can draw a conclusion that the conformations of the examined compounds, namely, the preferable existence of non-eclipsed *gauche*- and *trans*-forms with propeller arrangement of the pyridyl radicals relative to the P = Y bond (Y = lone pair, O, S, Se) fit into the overall conformational picture of the structure of the compounds of tri- and tetra-coordinated phosphorus with alkyl and thioalkyl radicals [1]. In all cases, these phosphines have non-eclipsed *gauche*- and *trans*-forms with propeller arrangement of the pyridyl as well as the alkyl radicals about the P = Y bond (Y = lone pair, O, S, Se).

Besides theoretical value, the results of the present study can be useful for prediction and rationalization of reactivity of tris(2-pyridyl)phosphine and its chalcogenides, for example, in investigation of their coordination properties with respect to the metals of different nature.

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